journal
J. Am. Ceram. Soc., 72 [7] 1249-52 (1989)

Grain-Size and *R*-Curve Effects in the Abrasive Wear of Alumina

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Results of sliding wear tests on three alumina ceramics with different grain sizes are discussed in the light of crack-resistance (R-curve, or T-curve) characteristics. The degree of wear increases abruptly after a critical sliding period, reflecting a transition from deformation-controlled to fracture-controlled surface removal. This transition occurs at earlier sliding times for the aluminas with the coarser-grained microstructures, indicative of an inherent size effect in the wear process. A simplistic fracture mechanics model, incorporating the role of internal thermal expansion mismatch stresses in the crack-resistance characteristic, is developed. The results suggest an inverse relation between wear resistance and large-crack toughness for ceramics with pronounced R-curve behavior. [Key words: alumina, R curve, wear, brittle materials, grain size.]

The brittleness of ceramics can lead to rapid wear by microfracture in severe local-contact conditions. Erosion by particle impact[1] and surface removal by machining[2] are two documented cases in point. Theoretical treatments of contact fracture mechanics[3] indicate that the associated wear rates should be greatest for materials with the least toughness, T.[§] The experimental erosion evidence,[1] embracing a broad range of brittle ceramics, lends some support to this contention. However, the growing realization that the toughness of a given material can be a sensitive function of the crack size (T-curve, or R-curve behavior),[4–8] depending strongly on the microstructural makeup, has led to some contradictory conclusions. Indeed, from machining tests on alumina ceramics that differ only in microstructure, it appears that the machining rate actually *increases* with the toughness,[2] at least with the toughness measured in traditional large-crack test specimens. Accordingly, when specifying toughness parameters for ceramic materials in the context of wear properties, it has become necessary to make due allowance for the microstructural scale of the fracture process.

In this work we present some new data on microfracture-associated abrasive wear of alumina ceramics in which grain-size effects are apparent. We show that it is necessary to consider specific details of material removal in relation to the underlying mechanisms that determine the T-curve behavior. We also show that the resistance to fracture-associated surface removal in these materials is least for alumina materials with the greatest large-scale toughness, i.e., in the coarser-grained materials. Following an earlier hypothesis by Rice and co-workers,[9,10] we argue that this seemingly contradictory relation between wear resistance and traditional toughness parameters is linked to the presence of residual stresses in individual grains, and that the influence of these stresses on the contact-fracture process is manifest as a microstructural size effect.

Accordingly, alumina specimens for wear testing were prepared by hot-pressing. The starting material was ultra-high-purity alumina powder[¶] doped with magnesia solute additive. Aluminas with three grain sizes, ℓ=4, 8, and 20 μm (as measured by a lineal intercept method[11]), were fabricated by suitable adjustment of the hot-pressing cycle.

The corresponding wear data are shown in Fig. 1. (These data represent just part of a broader, systematic study in our laboratories of wear transition behavior in ceramics, to be presented elsewhere.[12]) The test geometry for the data in Fig. 1 is that of a rotating sphere (silicon nitride, 12-mm diameter, 450-N applied load, 100 rpm) on three flat specimens (alumina). The three specimens are aligned with their surface normals in tetrahedral coordination relative to the rotation axis of the sphere and are mounted onto a bearing assembly to ensure equal distribution of the applied load. All tests were run at room temperature with purified paraffin oil as a lubricant. Wear is quantified by the ensuing scar diameter on the alumina specimen surfaces. Figure 1 shows that the scar diameter increases monotonically with sliding time. In the initial stages, this increase is relatively slight, indicative of a deformation-controlled removal process, and is independent of grain size. At a certain critical sliding time, however, the scar diameter for each material increases abruptly; moreover, the larger the grain

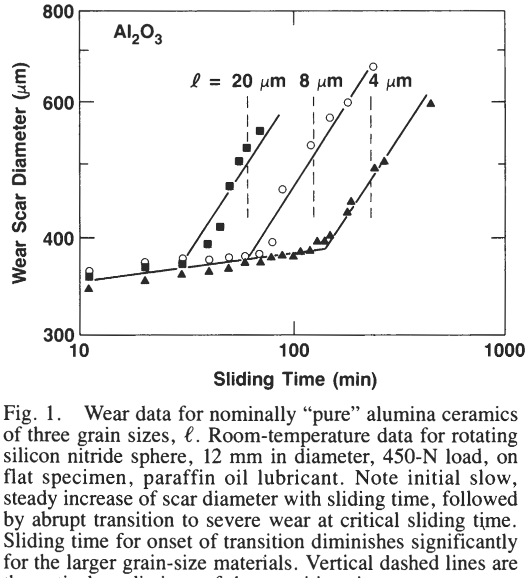

Fig. 1. Wear data for nominally "pure" alumina ceramics of three grain sizes, ℓ. Room-temperature data for rotating silicon nitride sphere, 12 mm in diameter, 450-N load, on flat specimen, paraffin oil lubricant. Note initial slow, steady increase of scar diameter with sliding time, followed by abrupt transition to severe wear at critical sliding time. Sliding time for onset of transition diminishes significantly for the larger grain-size materials. Vertical dashed lines are theoretical predictions of the transition times.

R. W. Rice—contributing editor

Manuscript No. 198831. Received October 20, 1988; approved December 5, 1988.
Supported in part by the U.S. Air Force Office of Scientific Research and by the Gas Research Institute. The work of S-J. Cho was supported by the Korea Science and Engineering Foundation.
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§The crack-size-dependent "toughness" (T) is alternatively referred to as K_R in the fracture mechanics literature and is related to the "crack resistance" (R) by $T=(RE)^{1/2}$, with E as Young's modulus.
¶AKP-HP powder, 99.99% pure, 0.5-μm crystallite size, Sumitomo Chemical America, Inc., New York, NY.

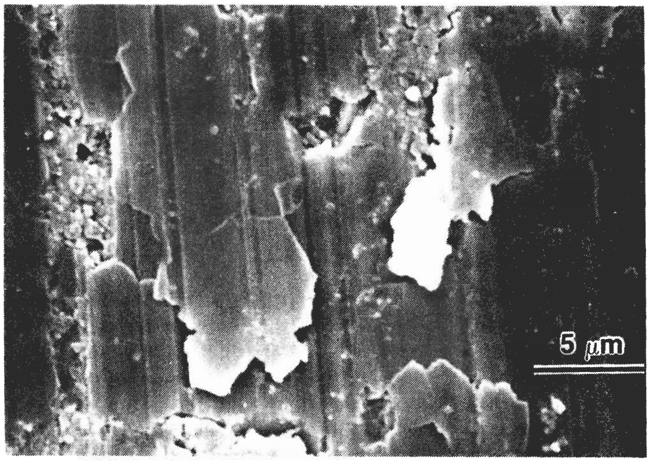

Fig. 2. Scanning electron micrograph of the wear surface of 4-μm-grain-size alumina after transition. Note extensive cracking at the grain boundaries.

size, the earlier this transition occurs. The coarser material is clearly more susceptible to severe surface degradation.

Examination of the specimen surfaces indicates that the abrupt transition to severe wear is associated with the onset of grain-localized microfracture. The scanning electron micrograph in Fig. 2 illustrates the appearance of the wear surface of the 4-μm-grain-size alumina after the transition. Before the transition, the damage shows only smooth scratches, typical of a deformation-controlled wear process. In Fig. 2, on the other hand, we see additional, severe damage in the form of extensive cracking along the grain boundaries, with dislodgement of individual grains and associated wear debris.

Transmission electron microscopy provides further clues to the transition mechanism. Figure 3 shows micrographs of foils (prepared by thinning from below the test surface[13]) of the same alumina as in Fig. 2 outside and inside the worn area just after the onset of transition. The pristine material in Fig. 3(A) shows a relatively "clean," i.e., dislocation-free, microstructure. Note the presence of void-like defects (inherent porosity after hot-pressing) at many of the triple points of the grain-boundary structure. The worn material in Fig. 3(B) shows severe accumulation of dislocation pileups and twins with strong crystallographic features, characteristic of abrasion damage in ceramics.[13] At the same time, the triple-point defect structure has apparently evolved into an interconnected grain-boundary microcracking pattern. Note also the presence of diffraction bend contours, indicative of substantial damage-induced residual stress in the foil.

To explain the results, we need to elaborate on the nature of the toughness versus crack-size function (the T curve) for alumina. In Fig. 4 we plot this functional dependence for the three grain sizes investigated in Fig. 1, using computations from an independent study of the toughness properties of alumina ceramics.[14] The underlying basis of these plots is a model for increasing crack resistance, in which the grain-boundary crack is initially unimpeded over facet dimensions, but that as extension proceeds the interface is increasingly bridged by restraining, interlocking grains. (There is strong physical justification for this model from direct observations of crack growth in alumina and other ceramics.[5,8]) Key to the bridging process is the presence of internal (thermal expansion mismatch) stresses in the noncubic alumina matrix:[14] at small crack size, c, the microcracks tend to propagate preferentially in regions where these internal stresses are tensile, effectively reducing the intrinsic, grain-boundary toughness. At large c, the compressive component of the internal stress leads to increased crack restraint via a dominating influence on Coulombic frictional tractions that resist pullout of bridging grains. From the fact that the final toughness exceeds the initial toughness in the plots of Fig. 4, we see that the deleterious effect of the internal stresses at low c is more than outweighed by the countervailing influence of the bridging elements at large c. Now the magnitude of the internal stresses ($\sigma_I \approx E\Delta\alpha\Delta T$, where E is Young's modulus, $\Delta\alpha$ is the differential expansion coefficient, and ΔT is the quench temperature range) *is independent of the grain size.* The tendency for the curves in Fig. 4 to cross each other is therefore associated with an inherent spatial scaling effect, determined ultimately by the crack extension distance between restraining bridges; the bridging distance is in turn proportional to the grain size.[14] Thus, in the region of greatest pertinence to wear processes (i.e., small c), the resistance to crack extension for any given material tends to its minimum value; moreover, this minimum is

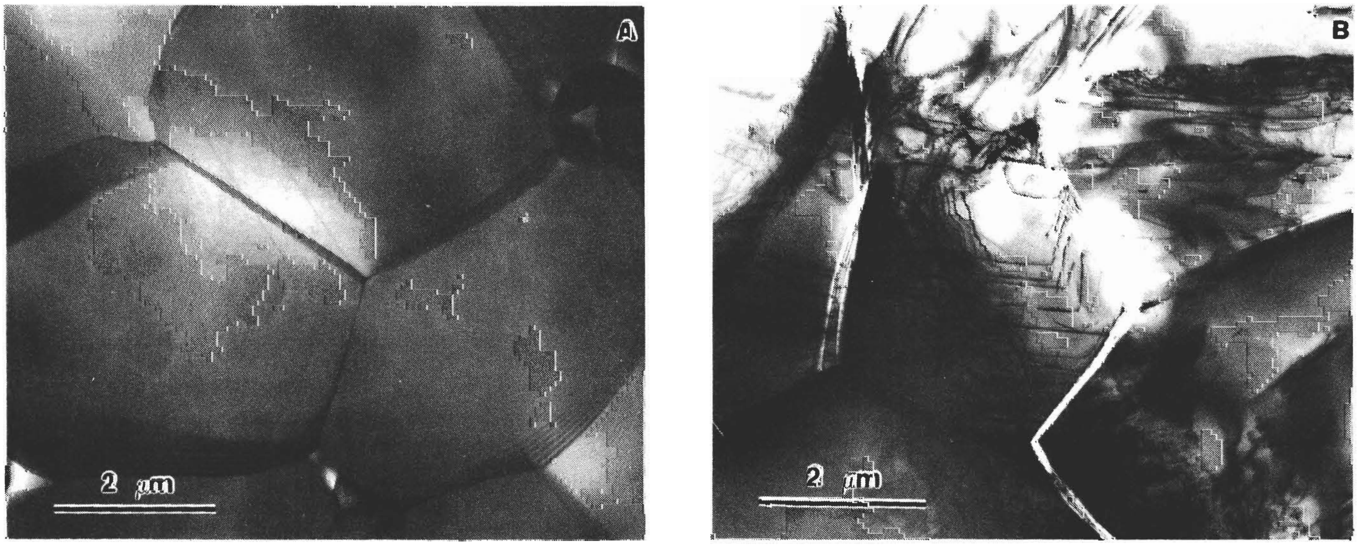

Fig. 3. Transmission electron micrograph of same alumina in Fig. 2: (A) outside and (B) inside wear scar. Accumulation of severe contact-damage stresses are responsible for propagating the flaws (prepresent triple-point defects evident in (A) or subsequent damage-induced defects) to form grain-boundary microcracking pattern.

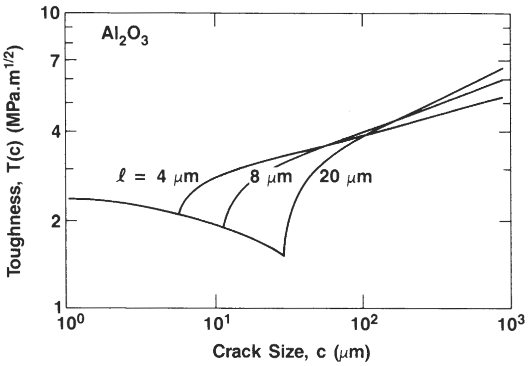

Fig. 4. Toughness versus crack-size function (T curve) for the three grain-size aluminas investigated in Fig. 1. Curves calculated from toughness model based on a crack-interface bridging mechanism, from Ref. 14. Note tendency for curves to cross each other.

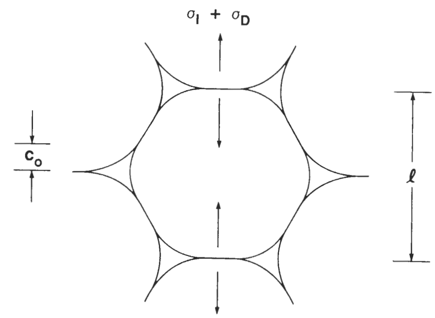

Fig. 5. Schematic of idealized grain structure, dimension ℓ, with (triple-point) boundary flaws, dimension c_0. Flaws were subjected to tensile components of internal stresses σ_I and damage stresses σ_D.

strongest in those materials with the maximum large-crack toughness. These trends are in essential agreement with those reported by Rice and co-workers.[9,10]

With this background, we may develop a simplistic fracture mechanics description of the wear results. We suppose that there are grain-boundary flaws in the alumina, on the microscale, from which extended microcracking can generate. These flaws may either be prepresent, e.g., the triple-point defects mentioned, or be generated by the damage process itself, e.g., from stress concentrations at the (pileup) deformation elements in Fig. 3(B). We do not attempt to distinguish fine details of the flaw geometry here, assuming only that the size of these flaws scales with the grain diameter. We now assert that the critical condition for the onset of local microfracture in the wear experiment is one in which the damage-induced tensile field causes the larger flaws to become unstable. This assertion is given credence by the well-known phenomenon of *spontaneous* microfracture in ceramics at a critical grain size under the action of the *internal* (thermal expansion mismatch) tensile field alone.[15] We envision the damage stresses as *augmenting* the preexisting stresses, effectively lowering the grain (flaw) size at which the critical state is achieved.

To quantify this description, consider the driving forces on the flaws in the grain configuration in Fig. 5. We take the initial flaw size to be proportional to the grain dimension, $c_0=\beta\ell$, where β is a scaling coefficient. Then we may write the equilibrium condition for the flaws in terms of superposed stress-intensity factors

$$K_I+K_D=T_0 \tag{1}$$

where T_0 is the intrinsic grain-boundary toughness and the subscripts I and D relate to the internal and damage-induced tensile stresses σ_I and σ_D, respectively. Neglecting gradients in the stress distributions over the flaw dimensions, the equilibrium requirement may be expanded in the form[16]

$$\psi\sigma_I(\beta\ell)^{1/2}+\psi\sigma_D(\beta\ell)^{1/2}=T_0 \tag{2}$$

where ψ is a crack-geometry coefficient $(2/\pi^{1/2})$ for penny-shaped cracks. The equilibrium in Eq. (2) is unstable and may be achieved at either critical ℓ or critical σ_D.

Critical $\ell=\ell_*$ corresponds to spontaneous microfracture at $\sigma_D=0$: this relation provides us with a convenient bounding condition for Eq. (2):

$$\psi\sigma_I(\beta\ell_*)^{1/2}=T_0 \tag{3}$$

For alumina ceramics, independent, approximate estimates of the grain-boundary toughness, critical grain size, and intensity of internal stresses give T_0=2.1 MPa·m$^{1/2}$ (Ref. 17), ℓ_*=400 μm (Ref. 15), and σ_I=100 MPa (Ref. 18), respectively. From Eq. (3) we estimate that β=0.8, corresponding reasonably to an initial flaw size on the order of the grain diameter. Substituting Eq. (3) into Eq. (2) allows us to determine the critical σ_D condition as a normalized function of grain size, thereby circumventing any uncertainty in this estimate of β arising from sensitivity of Eq. (3) to T_0, ℓ_*, and σ_I:

$$\sigma_D(\ell)=\sigma_I[(\ell_*/\ell)^{1/2}-1] \tag{4}$$

We note that Eq. (4) has something of a Hall–Petch $(\ell^{-1/2})$ relation, but with the spontaneous microfracture condition as a natural upper limit.

Equation (4) may now be used to determine the magnitudes of the damage-induced stress necessary to cause wear-associated grain spalling. Table I shows the calculated values for the grain sizes pertinent to the data in Fig. 1. Note that these stresses are substantial, characteristic of the severe deformation levels that attend typical point-contact (indentation) events[13,19,20] in brittle solids. These critical stresses diminish with increasing grain size, consistent with the observation in Fig. 1 that less sliding time is required to induce the transition in the coarser aluminas. If we postulate that the damage stresses accumulate at a constant rate, $\dot{\sigma}_D=\sigma_D/t$, in the wear process (a postulate supported by the steady, grain-size-independent increase in scar diameter in the initial stages of wear in Fig. 1), then we may make quantitative predictions of the trends for the transition regions. The vertical dashed lines in Fig. 1 correspond to such predictions for a stress-accumulation rate $\dot{\sigma}_D$=5 MPa·s^{-1}.

Our simplistic model is able to account for the major grain-size-related transition effects in wear data in alumina ceramics. The implications concerning optimization of microstructures for maximum wear resistance are clear enough here — refine the grain size and, if possible, avoid internal stresses. We reiterate that such measures may run entirely counter to the requirements for maximum large-crack toughness (Fig. 4), so that ultimate material design may involve subtle compromise.

Table I. Wear-Damage-Induced Stress Levels Required to Cause Grain-Boundary Microfracture in Alumina Ceramics

Grain size, ℓ (μm)	Wear-damage stress, σ_D (MPa)
400	0*
20	347
8	607
4	900

*Spontaneous microfracture limit.

Acknowledgments

The authors thank S. M. Hsu for encouraging this work and R. W. Rice for discussions.

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